9865

~~This report has been prepared for information and record purposes and is not to be referenced in any publication.~~

NATIONAL BUREAU OF STANDARDS REPORT

9865
(Revised)

INTERLABORATORY COMPARISON OF THE POTENTIAL HEAT TEST METHOD

NBS

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT		NBS REPORT
4212229	(August 26, 1968) May 1, 1969	9865 Revised

INTERLABORATORY COMPARISON OF THE POTENTIAL HEAT TEST METHOD

by

D. Gross and M. G. Natrella

NBS

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

INTERLABORATORY COMPARISON OF THE POTENTIAL HEAT TEST METHOD

by

D. Gross and M. G. Natrella

ABSTRACT

Quantitative measurements of the total heat release by selected building materials were made during an interlaboratory study of the Potential Heat Test Method. Seven of the eleven participating laboratories ranked the five materials in the same order, and a single ranking change for three other laboratories would yield identical rankings. The general magnitude of within-laboratory repeatability and between-laboratory reproducibility for composite materials of generally low potential heat are indicated by statistical analysis of the results.

Results are reported on the effect of the amount of combustion promoter used and on differences in the first and second phase values. A discussion is presented on the effects of material sampling and on certain features of the experimental procedure which require special care. A tentative test method standard, containing complete details of the test procedure, is included as an appendix.

Key Words: calorimetry, combustibility, fire tests, heat of combustion, interlaboratory tests, oxygen bomb, potential heat

Interlaboratory Comparison

of

The Potential Heat Test Method

by

D. Gross and M. G. Natrella

1. Introduction

In March 1965, an interlaboratory comparison study of the potential heat test method was initiated by a Task Group of Subcommittee V (Definitions and Nomenclature) of ASTM E-5 (Fire Tests of Materials and Constructions). One goal of the Task Group was to evaluate the suitability of the test method for measuring and classifying materials according to their degree of combustibility.

The test method had been developed several years earlier [1] to measure the total heat released under typical fire exposure conditions without regard to the rate at which the heat was released. The method makes use of standard calorimetric techniques in which the burning of small quantities of combustible in an otherwise inert material is assured by use of a combustion promoter which is added prior to test. By measuring heat of combustion in an oxygen bomb calorimeter both before and after exposure to a "standardardized fire" (2 hr in air at 750 C), the difference may be considered as the potential heat of the material. (See Appendix A)

The Task Group consisted of the following members:

Mr. I. A. Benjamin, Granco Steel Products Co., Chairman
Mr. P. E. Baseler, Building Officials Conference of America
Mr. D. Gross, National Bureau of Standards
Dr. J. R. Jutras, National Research Council of Canada
Mr. J. A. Wilson, Factory Mutual Engineering Corporation*

The NBS Fire Research Section mailed specimens to the participants in May 1965 and prepared a preliminary report of the test results in June 1966. A conclusion of that report was that a higher degree of variability existed among the nine participating laboratories than would be expected of a single laboratory performing standardized oxygen bomb calorimeter determinations of the heat of combustion. However, the majority of laboratories tended to rank materials in the same order, or in an order which could be made the same by a ranking change of a single material. The initial report also suggested that more specific test instructions and evaluations be provided in future test programs relating to material sampling, to the proportion of combustion promoter, and to controls over pulverization, weighing and temperature measurement.

* Deceased

A second phase of the interlaboratory comparison of the potential heat test method was initiated in July 1966, incorporating the knowledge gained from the initial phase. In addition to prescribed revisions to the test method supplied to the participants, the second phase was designed to permit evaluation of the percent combustion promoter, agreement of results of duplicate determinations within each laboratory (repeatability), and agreement of results between laboratories (reproducibility). The test results were returned to NBS for evaluation in the period, September 1966 to May 1967, and are summarized in this report.

2. Participants

A total of eleven laboratories, including the original group of nine, participated in the second phase study. These were:

Ohio State University
Underwriters Laboratories, Inc.
U. S. Forest Products Laboratory
Fire Research Section, National Research Council of Canada
Johns-Manville Fiberglass Division
Owens-Corning Fiberglas Corporation
Factory Mutual Engineering Division
U. S. Bureau of Mines
Robert W. Hunt Company
Benjamin Foster Company
National Bureau of Standards

The laboratories were identified by code letter only, and the letter S was given to the NBS laboratory.

3. Test Procedure

Details of the test procedure were described in the original paper [1] and in a tentative test method standard (Appendix A), distributed to the participants. Also distributed were the test method revisions (Appendix B), supplementary notes and instructions, and a summary data sheet. The instructions requested that a total of 12 tests be performed on five materials in accordance with the plan shown in table 1.

4. Materials

Also listed in table 1 are the test materials comprising five of the eight materials employed in the initial phase. The test materials, which are typical composite materials used in buildings at the present time were designated by code letter only, and no producer identification was intended or implied. With one exception, the original group of nine participating laboratories used the same piece of material remaining from the first phase. The 3- by 12-inch samples had been cut from larger sheets in a prearranged manner and a record kept of the sampling locations. See figure 1.

5. Results

Table 2 summarizes, for each laboratory, the individual and average values of the heat of combustion of the specimen (direct), the heat of combustion of the residue (after simulated fire exposure), and the potential heat, using 50 percent combustion promoter. Also listed is the median value of the set of eleven laboratory averages for each material.

Table 3 summarizes the percent residue values for all materials and laboratories, and illustrates the good agreement of this determination. For the five materials of Phase 2, the average percent residue values did not differ from the Phase 1 averages by more than 0.7 percent.

Table 4 lists, for each laboratory, average values of test parameters which affect the direct measurement of the heat of combustion. From this list, and the individual values on which they were based, the following points were noted:

1. The incremental temperature readout varied from 0.005 ^{o}F (as specified) to 0.04 ^{o}F (0.02 ^{o}C). For an average calorimeter water equivalent of nearly 2500 Btu/lb ^{o}F, based on a 1 gram sample*, a change of 0.04 ^{o}F corresponds to a change of 100 Btu per lb.

2. The average fuse wire correction ranged from 15 to 29 Btu/lb and the average acid titration correction, excluding material "C", ranged from 1 to 30 Btu/lb; the average acid titration correction for material "C" ranged from 9 to 187 Btu/lb, all values based on a 1 gram sample. Since material "C" contained approximately 16% sulphur, an appreciable correction was to be expected.

One laboratory separated composite materials "C" and "E" into their two-layered components and tested each component separately. The contributions of heat were then combined in proportion to their original weight fractions.

---

* Water equivalent is the number that is multiplied by the temperature rise and divided by the sample weight in grams to give the heat of combustion in Btu/lb. Also see footnote, table 4.

1 British thermal unit (Btu) = 1055 joules (J).

6. Statistical Analysis

6.1. Averages and Deviations

Because of the variety of materials tested and the variability of the results, each laboratory's results were considered as deviations from the median value of all laboratories for each material. The median value is the value such that half the results are smaller than, and half the results are larger than the median, and is less influenced by extreme or outlying values than is the arithmetic mean. The value for each laboratory and each material is the average of duplicates shown in table 2.* The median of all laboratories for each material, and the deviations from the median, are shown in table 5.

In a more detailed analysis of the data, laboratory U was omitted from many of the calculations because of extreme variability (see section on Variability). The median values omitting laboratory U are shown in the last column of table 5. However, the deviations shown are from the median for all 11 laboratories.

6.2. Ranking of Materials

A simplified overall summary of the interlaboratory comparison may be obtained by the comparison of material rankings listed in table 6. Seven of the eleven laboratories ranked the five materials in the same order, corresponding to the overall consensus. Furthermore, a ranking change of a single material would bring the other four laboratories into agreement with the consensus.

6.3. Variability

The range of duplicates for each laboratory and each material is shown in table 7. The average range of duplicates for each material (averaged over all laboratories) and for each laboratory (averaged over all materials) is also shown.

Consideration of this average range for the different laboratories indicated that the results for laboratory U should be omitted because of extremely large variability, particularly on materials "B" and "C" The last column of table 7 shows the average range for each material (averaged over all laboratories) excluding laboratory U.

---

* Throughout this report, the word duplicates refers to results from duplicate specimens.

The average range of duplicates is roughly the same size for materials "A", "C", and "D", and is two or three times as large for materials "B" and "E" in both the Direct and Potential Heat measurements. In the Residue, the average range is about the same size for all materials except "E", which is about twice as large.

Since materials "B" and "E" have higher mean values, as well as larger variability (true only of material "E" in Residue), one must consider the possibility that the variability increases with the mean value. Plots of the average of duplicates versus the range of duplicates (i.e. one point for each laboratory and each material) were made for Direct, Residue, and Potential Heat measurements in figures 2A, 2B and 2C. On the three plots, each material is identified by a different symbol. The scatter of points for materials "B" and "E" in the plot for the Direct (fig. 2A) indicates that, for these materials, there may be two levels of variability corresponding to two groups of laboratories rather than a general increase in variability with increasing mean value. This, in turn, raises the question of whether one group of laboratories was simply "more careful" than the other group, or whether there is some aspect of the test procedure which is interpreted or applied differently by different laboratories, i.e., a real difference in the method as actually used. Because of the nature of materials "B" (mineralized wood) and "E" (protected steel), there may be a problem in obtaining a homogeneous sample (see sections on Sampling and Experimental Procedures).

In the plot for Residue (fig. 2B) only material "E" seems to be possibly different in variability from the other materials. The plot for Potential Heat (fig. 2C) shows a more irregular pattern with more overall scatter than either the Direct or the Residue, as would be expected, because Potential Heat is calculated from the other two.

In order to be able to state the precision of a test method, it must be demonstrated that all laboratories (considered qualified to make the test) have the same within-laboratory variability. When this is not true for the whole collection of results from an interlaboratory test program, certain kinds of data screening may be indicated, or it may be necessary to make some refinement of the test procedure itself. Screening of the data might consist of eliminating certain laboratories whose variability was substantially larger than that of the other laboratories, or might require restricting the applicability of the conclusions to a certain range or kind of material.

In the present case, it was decided to perform an analysis of variance on the results from ten laboratories and three materials and, therefore, the precision statements below apply only to materials "A", "C", and "D". The analysis of variance yielded the following values:

	Standard Deviation for Repeatability 1/	Standard Deviation for Reproducibility 2/
	Btu/lb	Btu/lb
Direct	74	212
Residue	70	217
Potential Heat	92	243

1/ Calculated from duplicates within a laboratory.
2/ Including components due to differences between laboratories.

A convenient way of comparing results is by plotting the deviation from the median of the potential heat value from one test on the X-axis, and the deviation of the duplicate potential heat measurement on the Y-axis. In this arrangement, sometimes called a Youden plot [2], each pair of results from each laboratory is represented by a plotted point. The origin of the coordinate axes is located at the median value for that material and the graph is divided into four quadrants. Points tend to be equally distributed in all four quadrants when only random errors of measurement are present. Points tend to concentrate in the upper right and lower left quadrants when there are systematic biases by individual laboratories, i.e. laboratories tend to get high results on both duplicates or low results on both duplicates. As shown on figure 3, there is a tendency for points to concentrate in these two quadrants which is typical for most interlaboratory data.

If there were no biases, about 90% of the points should be within a circle whose radius is 2.15 times the standard deviation. For each material, several points (laboratories) lie outside such a circle; for materials "B" and "E", the circle is not appropriate and is omitted.

The band marked by the dashed lines is formed by two lines drawn tangent to the circle and parallel to the 45^{o} line through the origin. Points which lie outside the circle but inside the band represent laboratories which show a systematic deviation from the consensus, but whose errors of measurement are not excessive.

It should be added that several transcription errors in the data reported were noted and corrected after verification with the laboratory involved. It is possible that other errors of this and related types still remain and contribute to the variability of the data reported.

6.4. Combustion Promoter

For materials "A" and "D", determinations were also made using 75 percent combustion promoter. Table 8 lists the differences between the single determination with 75 percent promoter minus the average of duplicates with 50 percent promoter for direct, residue and potential heat. Values have been omitted for Laboratory U, whose values were too variable, and for Laboratories P and Z, which used other percentages of combustion promoter (60 and 83.3%) than that prescribed. The average differences between 75 percent and 50 percent promoter for the direct, residue and potential heat values on material "A" were +87, +124 and -10 Btu/lb, respectively. For material "D", the corresponding values were +134, +13 and +150 Btu/lb. Although higher heats of combustion were generally obtained with 75 percent promoter, the differences were not generally significant.

6.5. Phase 1 vs. Phase 2

Table 9 shows the difference, Phase 1 (1966) minus Phase 2 (1967), in heats of combustion (direct, residue and potential heat) for each material and each laboratory. Laboratories P and W are missing, since they did not participate in the Phase 1 tests, and Laboratory U was omitted as before. From the average differences for each material, there were no appreciable shifts in the interlaboratory average values compared to the first phase.

7. Discussion

7.1. Sampling

Several laboratories questioned whether the sample was too small to be representative of the heterogeneous engineering materials used. For the second phase, a sample of at least 1/2- by 3-inches and weighing at least 10 g was specified. Also, the original manner of selecting specimens for distribution to the participating laboratories had been such as to minimize differences due to possible material variations or nonuniformities within a manufactured sheet. (See figure 1).

A measure of the extent of this problem might be gaged by (a) variability in the direct bomb heat of combustion values, and (b) variability in the percent residue values for the muffle furnace specimens, since this is a measure of the combustible and volatile content.

From figure 2A and table 7, it may be noted that greater variability appears to be associated with materials "B" (mineralized wood) and "E" (protected steel) than with the other three materials. For these two materials, there appears to be a suggestion of separation into two groups at different heat of combustion levels. A study of this distribution with respect to the sampling arrangement of figure 1, does not suggest any obvious correlation between the measurement differences noted and the location of the individual samples on the original sheet. On the other hand, it appears possible that this characteristic variability may be associated with a differential separation of the components during specimen preparation by some laboratories. In this regard, it is interesting to note that the intentional separation of composite materials "C" and "E" into their layered components, and their separate testing by laboratory V, yielded consistent and meaningful heat of combustion values when their heat contributions were combined in proportion to their original weight fractions. Laboratory Z employed a similar separation procedure for material "E" and also obtained consistent results. This material presented difficulties in the preparation of a homogeneous sample to many laboratories.

As noted previously, there was good reproducibility in the percent residue values for all materials and laboratories. This suggests that variations among samples was not a very serious problem in this study. However, there are advantages to sampling from several locations on a given sheet and averaging the results. Such a procedure could well be included as part of the test procedure, particularly for grossly heterogeneous materials.

7.2. Experimental Procedures

It seems fair to state that although the test method involves essentially routine laboratory techniques, careful attention must be given to the detailed procedures. The slight systematic biases noted previously suggest that the test procedure may be subject to individual modifications that have a measurable effect upon the results. Nevertheless, it is clear that the test method need not be restricted to a few laboratories, and that valid results may be expected of any laboratory with the necessary equipment and qualified personnel.

Accurate temperature measurement is the essence of meaningful bomb calorimetry. The high degrees of variability noted in this study can be ascribed in large measure to the relaxation of this requirement by some of the participating laboratories.

The characteristic variability associated with materials composed of high and low combustibility fractions emphasizes the need for careful attention to the pulverization, sieving and mixing operations so as to avoid segregation or separation of components during specimen preparation.

Laboratory V made separate measurements on the two primary components of material "E" and then derived an overall potential heat measurement by combining the two results in proportion to the weight fraction of the constituents present in the laminate. They reported a potential heat of the coating of 5400 Btu/lb, and 2900 Btu/lb for the steel. The treatment of simple laminates in this way seems to provide one way of avoiding problems due to segregation of components.

The results of this study indicated a slight increase in heat of combustion when 75 percent instead of 50 percent combustion promoter was used, but the difference was not regarded as significant. However, the use of a higher proportion of combustion promoter may relieve possible doubts concerning the completeness of combustion.

Changes in the sampling procedure and in the pulverization, sieving and mixing operations have been suggested as revisions to the tentative test method (see Appendix B).

7.3 Precision

A conventional heat of combustion measurement, e.g., on coal, (10,000 to 15,000 Btu per lb) is expected that duplicates would agree within 0.3 percent within the same laboratory, and within 0.5 percent at different laboratories [3]. For liquid hydrocarbon fuels, acceptability of bomb calorimeter test results are specified in terms of the following limits [4]:

> "Repeatability - Duplicate results by the same operator shall be considered suspect if they differ by more than 55 Btu/lb.
>
> "Reproducibility - Results submitted by two or more laboratories should be considered suspect if they differ by more than 175 Btu/lb."

Since this test method requires the mixing of a combustion promoter with the specimen, as well as reporting the difference of direct and residue bomb calorimeter measurements and accounting for weight changes during the muffle furnace exposure, errors of somewhat greater magnitude are to be expected. Based on the measured variability associated with 3 materials and 10 laboratories, it would be expected that duplicate measurement of Potential Heat would agree within 200 Btu/lb within the same laboratory, and within 500 Btu/lb at different laboratories.

8. Summary

The experience gained from the interlaboratory comparison of the Potential Heat Test Method should prove valuable to those interested in quantitative and reproducible measurements of the total heat released from building materials under simulated fire conditions. A total of eleven laboratories participated and provided test results on five composite materials, all of which were of generally low potential heat as a critical test of the method. Based upon comments and suggestions from the participating laboratories, and a study of the test variations employed, the more important details of the test procedure were pinpointed for inclusion and/or additional emphasis in a test method revision.

Seven of eleven laboratories ranked the five materials in the same order and a single ranking change for three other laboratories would yield identical rankings. Two materials ("B" and "E") presented problems to some laboratories in the preparation of a homogeneous sample. Based on the other three materials ("A", "C" and "D"), the standard deviation for repeatability within a single laboratory of the Potential Heat measurement was found to be 92 Btu/lb, and the reproducibility (standard deviation including difference between laboratories) was 243 Btu/lb. Duplicate measurements of Potential Heat would be expected to agree within 200 Btu/lb within the same laboratory and within 500 Btu/lb at different laboratories.

9. References

[1] Loftus, J. J., Gross, D. and Robertson, A. F., "Potential Heat -- A Method for Measuring the Heat Release of Materials in Building Fires," ASTM Proc. 61, 13336-48 (1961).

[2] Youden, W. J., "Graphical Diagnosis of Interlaboratory Test Results", Industrial Quality Control, vol. XV, No. 11, May 1959.

[3] Standard Methods of Laboratory Sampling and Analysis of Coal and Coke, ASTM Designation D 271-64, 1966 Book of ASTM Standards, Part 19.

[4] Standard Method of Test for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter, ASTM Designation D 240-64, 1966 Book of ASTM Standards, Part 17.

Appendix A. Tentative Method of Test for

POTENTIAL HEAT RELEASE OF MATERIALS IN BUILDING FIRES

Scope

1. This method of test provides a means of determining the potential release of heat of materials involved in building fires. The method is applicable to a variety of materials including metals, and especially materials of low combustible content. Determinations may be made on simple materials, or on composite assemblies of materials from which a representative sample can be taken and pulverized into a homogeneous mixture.

Definition

2. Potential heat of a material is the difference between the heat of combustion of a representative sample of the material and the heat of combustion of any residue remaining after exposure to a simulated standard fire, using combustion calorimetric techniques.

Summary of Method*

3. One of two specimens removed from the material to be tested is pulverized, pelleted, and burned in a high-pressure oxygen atmosphere. The process is generally as described in ASTM D 271 (Laboratory Sampling and Analysis of Coal and Coke), but with certain modifications or permissible exceptions, to be noted in the test procedure. This determines the gross heat of combustion of the material. The second specimen is heated in air for 2 hr at a temperature of 1382 F (750 C), conditions adopted as representing a standard fire exposure. A portion of the resulting residue of this specimen, if any, corresponding to a predetermined weight of original material, is ground or pulverized, mixed with a combustion promoter, and pelleted for burning as was the first specimen. After correcting for the heat produced by the combustion promoter, the difference in heating values of the two specimens is the potential heat, as defined in par. 2. The test procedure is illustrated schematically in fig. A1.

Apparatus and Materials for Test

4. The apparatus and materials required for the test are listed below.

(a) Oxygen bomb calorimeter, including firing circuit and fuse wire.

(b) Muffle furnace (having small opening or port for passage of air supply tube).

* See Reference [1].

(c) Hand mill (or ball mill).

(d) Pelleting press.

(e) Microbalance, weighing to 0.1 mg.

(f) Oxygen cylinder and accessory equipment.

(g) Compressed air supply.

(h) Standard alkali solution.

(i) Combustion promoter, National Bureau of Standards standard material, sample 39 i, benzoic acid (calorimetric standard).

(j) Parts associated with muffle furnace firing (illustrated in fig. A2).

(1) Specimen container for use in the muffle furnace; a suitable part may consist of a fused silica or ceramic tube, 1-1/4 inches inside diameter by 4 inches long, closed at one end.

(2) Cap to fit open end of specimen container; cap to be provided with an opening to pass an air supply tube with loose fit.

(3) Air supply tube; may be of porcelain, fused silica, or corrosion-resistant metal; inside diameter 3/16 inch minimum; length to extend beyond port of muffle furnace.

(4) Wire specimen holder; of corrosion-resistant metal; formed to support the specimen away from the walls of the specimen container, for circulation of air about specimen.

(5) Support of fire brick or similar material shaped to hold the specimen container and its cap in alignment with the muffle furnace port, so that the air supply tube may be positioned in the specimen container.

Test Specimens

5. Two air-dry test specimens representative of the material or assembly involved are required for each determination. A specimen is considered "air-dry" when it has reached constant weight in an atmosphere maintained at 73 ± 2 F (23 ± 1 C) and 50 ± 5 percent relative humidity. If the test subject is an assembly or composite material, it is essential that the several elements of the subject

are contained in the test specimen in the same proportions that they occur in the subject. The two specimens are subjected to separate test procedures.

Procedure for Direct Bomb Test

6. (a) The specimen shall be pulverized, using a hand mill or other means as may be necessary, into a form suitable to pass a No. 60 sieve.

(b) A pellet, weighing approximately 1 g, shall be prepared from a representative sample of the powder, and then weighed.

Note 1: All weight measurements shall be to the nearest 0.1 mg.

Note 2: Pellets shall be made in accordance with the method for the particular pelleting press in use and of a size convenient for the specimen cup. The pellets shall be no harder than necessary to prevent their disintegration when fired.

(c) The prepared pellet is the test specimen for the procedure for determination of the heat of combustion in accordance with ASTM D271-64, par. 51-55 (oxygen bomb test). Also see ASTM E-144, Recommended Practice for Safe Use of Oxygen Combustion Bombs.

Note 3: Modifications and exceptions to the requirements of ASTM D271-64 are listed in the Addendum.

(d) If, after being fired in the oxygen bomb, the pellet is found to have burned completely, or to have left no significant amount of residue or ash, the heat of combustion on an air-dry basis may be computed, and the following three steps (e), (f), (g), shall be omitted.

(e) If the pellet does not burn, or a residue remains after the firing, another 1 g pellet is prepared, this time using an intimate mixture of the powdered sample and a standard sample of benzoic acid combustion promoter, in equal weight proportions.

(f) The pellet prepared with the added benzoic acid is used as the test specimen following the same procedures as for the original specimen.

(g) A correction for the heat of combustion of the benzoic acid present in the pellet is applied to the measured heat released by the specimen. The heat of combustion of the sample material, on an air-dry basis, is then computed.

Procedure for Muffle Furnace and Bomb Test

7. (a) An air-dry specimen representative of the test material or assembly shall be cut in the form of a rectangular prism 1/2- by 3/4- by 3-inches. Sheet materials may be folded or laminated to these dimensions.

(b) The muffle furnace is preheated to 1382 ± 18 F (750 ± 10 C). The specimen is weighed, and placed on the wire support 4. (j) (4) in the specimen container 4. (j)(1). The container is closed with its cap 4. (j)(2), and placed in the firebrick base 4. (j)(5) in the muffle furnace in such position as to align the muffle furnace port and the opening in the specimen container cap. The external air supply tube 4. (j)(3) is passed through the port into the container in proximity to the specimen. Firing is continued for 2 hr with a regulated air flow of 0.1 ft^3 per min, referred to 60 F and 30.0 inches Hg, supplied to the specimen. If ignition should occur immediately upon placing the specimen in the furnace, application of air shall be delayed until the initial flaming has stopped.

(c) The container with the specimen shall be cooled in a desiccator, after which the weight of the residue is determined.

(d) If the residue from the muffle firing procedure is less than 5 percent of the initial weight of the specimen, the following steps (e) and (f) are omitted, and the heat of combustion previously determined under the direct bomb test, par. 6 (d), shall be reported as the potential heat of the material.

(e) If the residue after the muffle firing is in excess of 5 percent of the original specimen weight, the residue shall be pulverized, mixed with an equal weight of benzoic acid and treated as specified in the procedure for direct bomb test to determine the heat of combustion (of the residue).

(f) To determine the heat of combustion of the residue per unit weight of original specimen, multiply the heat of combustion determined in par. 7. (e) above by the ratio of residue weight, 7. (c) to the original specimen weight 7. (b).

Potential Heat

8. The potential heat of a material is determined by subtracting the heat of combustion of the residue remaining from the muffle furnace firing, par. 7. (f), from the heat of combustion of the material

established in the direct bomb test, par. 6 (g). The potential heat is thus a measure of the heat released by a material in the muffle furnace firing, the conditions of which are considered to simulate a standard fire. For most materials, potential heat may be reported in heat units per unit weight, or where appropriate to the material and its use, it may be expressed on the basis of volume or surface area of the material. For materials such as metals where the combustion process is relatively slow, and is a function of surface area, potential heat should appropriately be reported on a surface area basis only.

ADDENDUM

Modifications and exceptions to the requirements of ASTM D 271-64 are as follows:

par. 52 (e) Fuse wire may be that provided for the bomb in use; a suitable correction factor for the wire shall be applied.

par. 53 (b) Benzoic acid is a suitable substance for standardization procedures (see 4. (i) of this standard).

par. 54 (a) Alternate method of Note 36 for materials of high ash content not used; in cases of incomplete burning, a combustion promoter is added (see 6. (e)).

par. 54 (f) An ignition system supplied for the bomb may be substituted.

par. 54 (h) Where materials leave a residue, remove the cup containing the residue, then proceed to rinse out the bomb and titrate as described in this paragraph.

par. 55 (Note 44) The method of this test for potential heat release of materials gives the gross heat of combustion of a material in an air-dry condition; net calorific value (net heat of combustion) calculations are not normally a part of this procedure. (see footnote*).

Replace existing sections in Tentative Test Method (Appendix A) with the following:

6. (a) The specimen shall be pulverized into a form suitable to pass a No. 60 sieve. Grinding, filing or milling operations, whether manual or mechanical, should be effected on the cross-section which is normal to the grain, fiber or other process-induced orientation. Particular care should be taken to avoid segregation or separation of components. The representative specimen shall not be smaller than 1/2 inch by 3 inches by the thickness as supplied, nor shall the resultant powder weigh less than 10 grams. For grossly, heterogeneous materials, a representative specimen should be obtained by combining samples of material from different units (or sheets) and from different locations on each unit.

Note: While many materials may be suitably reduced using a clean carbide double bastard file and/or mortar and pestle, it may sometimes be useful to (dry-ice) freeze materials containing asphaltic, mastic or plastic components prior to filing, or to use mechanical blendors, ball or hammer mills, grinders, milling or lathe cutters, etc. For laminated materials, it may be preferable to separate into component layers and to grind, file or pulverize each component separately. The powdered components may then be intimately mixed in proportion to their original weight fractions and the mixture tested, or, alternately, each component may be tested separately and the contributions of heat combined in proportion to their original weight fraction.

6. (b) A pellet, weighing approximately 1 g, shall be prepared from an intimate mixture of the powder, and then weighed.

6. (e) If the pellet does not burn, or a residue remains after the firing, another 1 g pellet shall be prepared using approximately 1/2 g portions of the powdered sample and a standard sample of benzoic acid combustion promoter. Weigh each portion accurately to 0.1 mg, mix together thoroughly and pelletize. Record the weight of the pellet to 0.1 mg. Any loss in weight after mixing and pelletizing should be subtracted from the sample and the combustion promoter in proportion to their original weight fractions, and the corrected weights used in the heat of combustion calculations.

Add the following after Note 3, section 6. (c)

Note 4: For tests on specimens which are predominantly metallic, the use of a silica combustion capsule is recommended. The water equivalent of the calorimeter using the silica capsule should be measured and used.

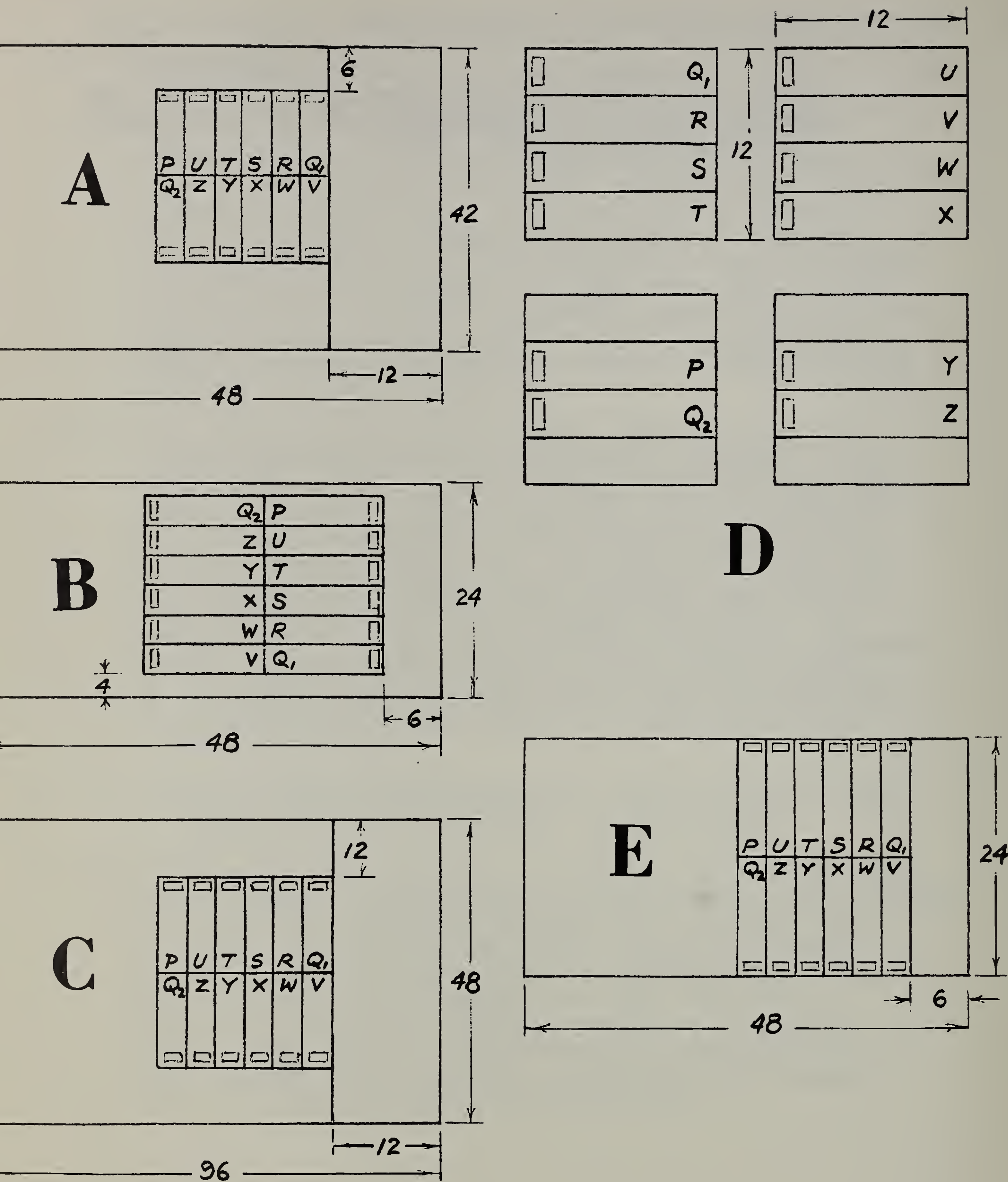

Fig. 1. Sampling Arrangement - Potential Heat Test Specimens

▭ = label

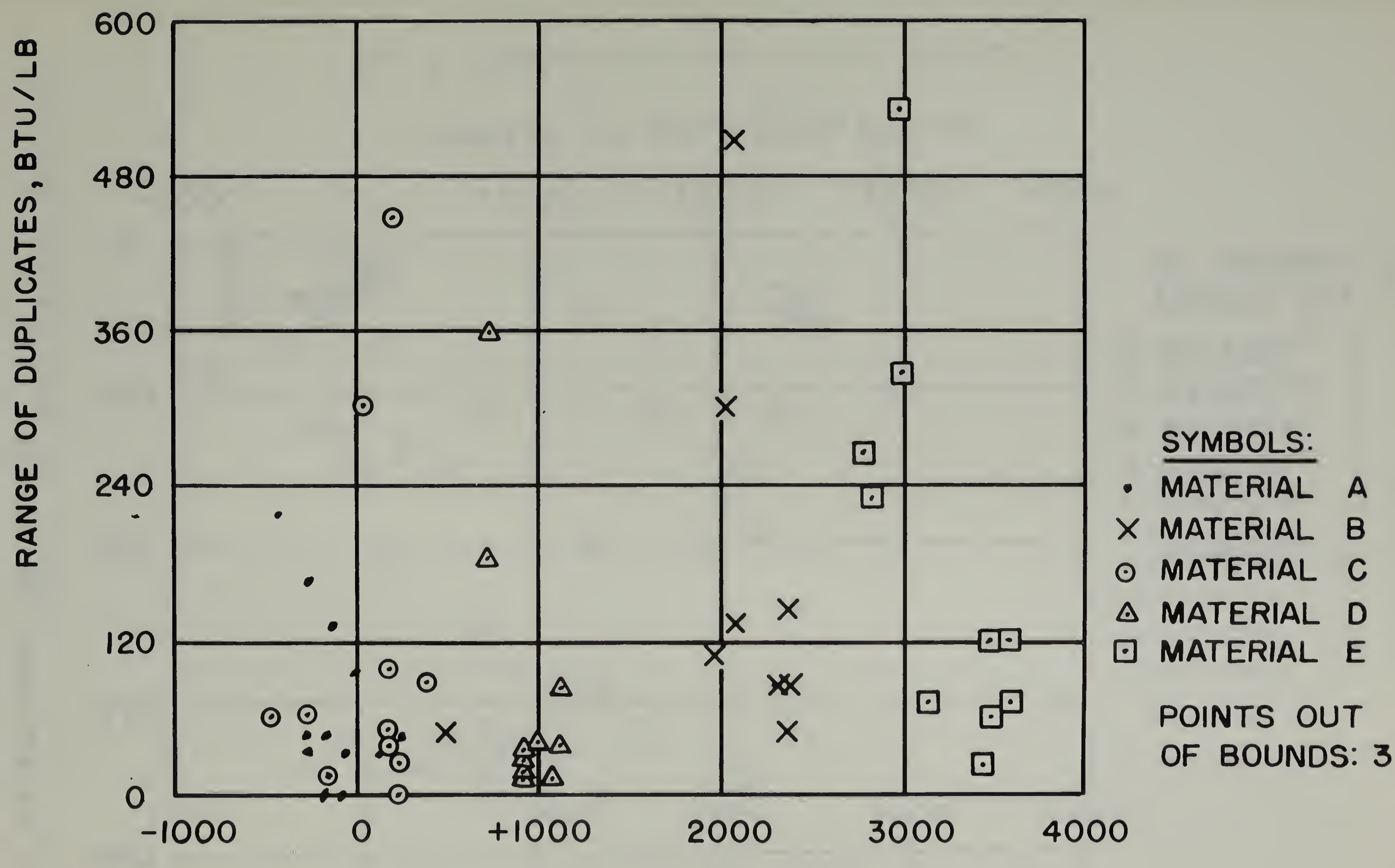

Fig. 2A Range of Duplicates vs Average - Direct

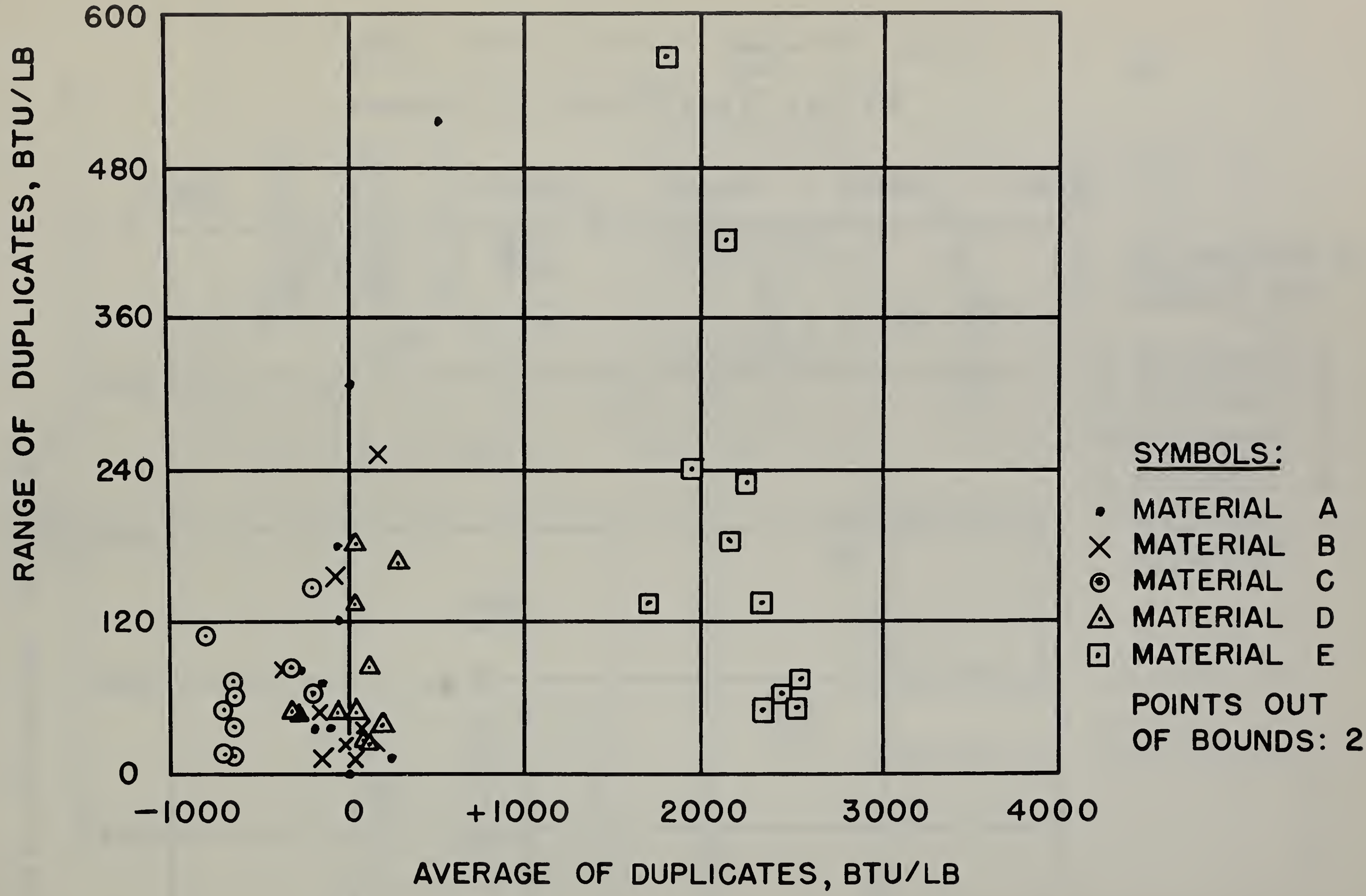

Fig. 2B Range of Duplicates vs Average - Residue

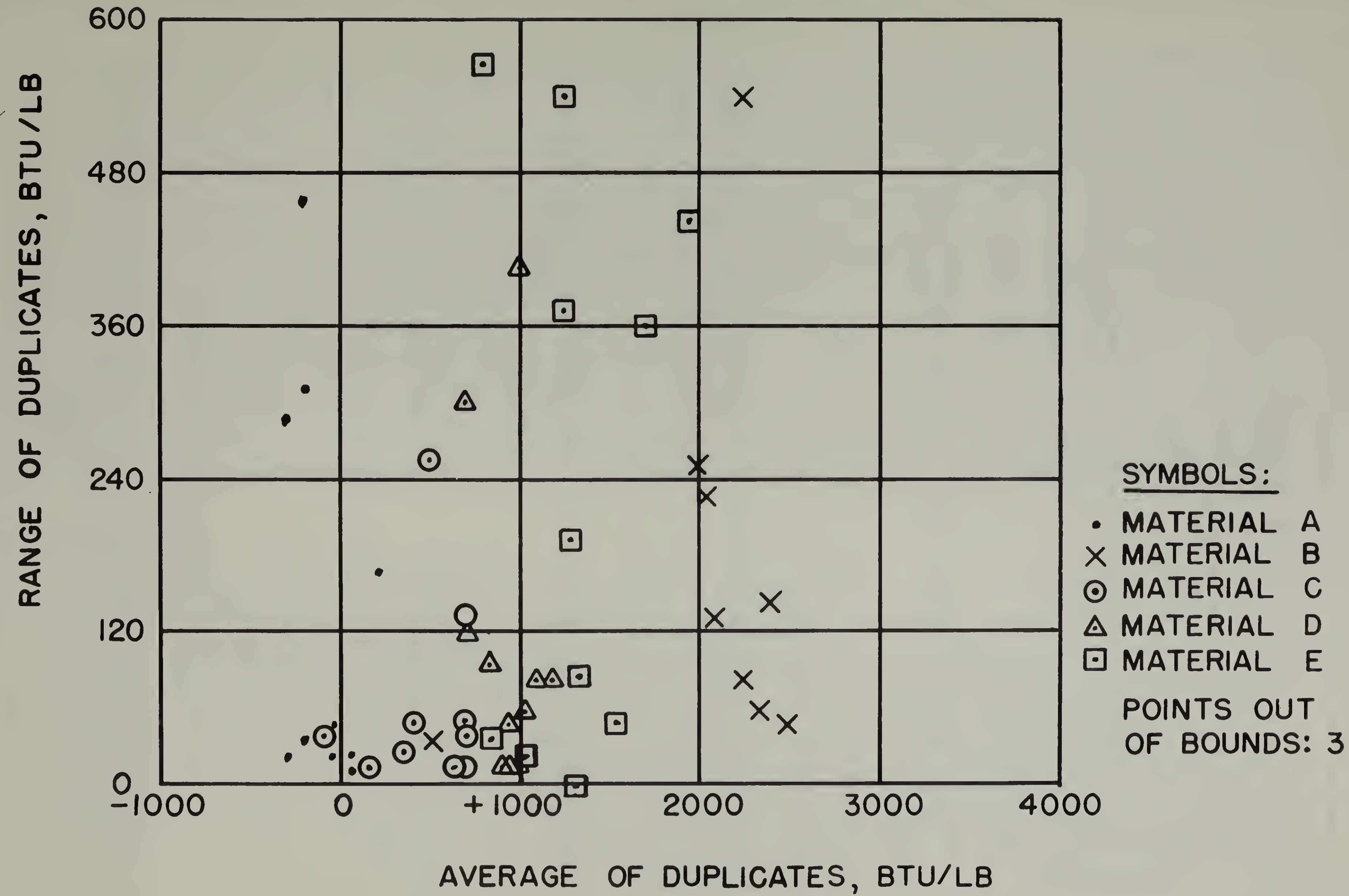

Fig. 2C Range of Duplicates vs Average - Potential Heat

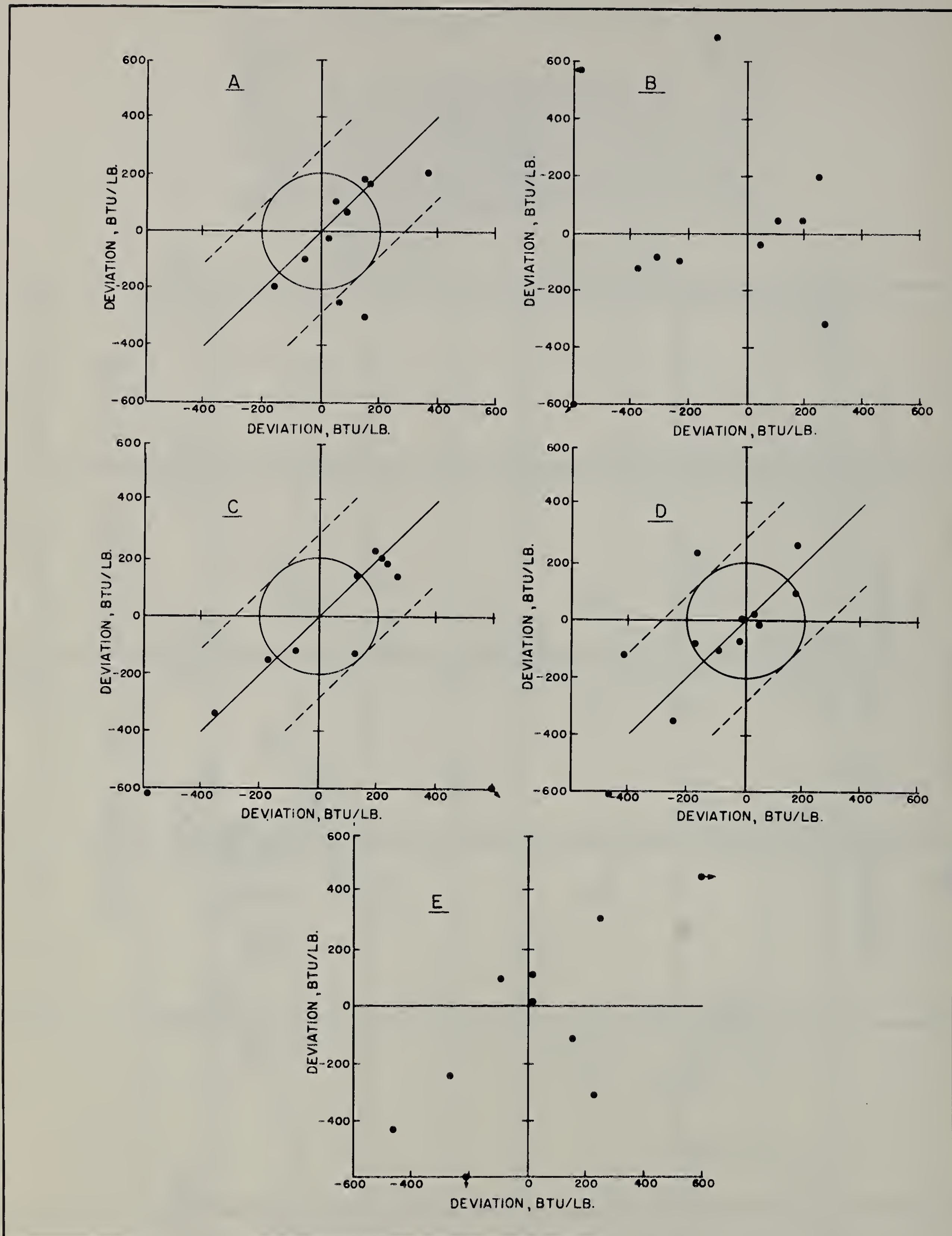

Fig. 3 Youden Plot of Duplicate Measurements of Potential Heat

Median	A:	- 112 Btu/lb
	B:	2258 Btu/lb
	C:	492 Btu/lb
Values	D:	984 Btu/lb
	E:	1288 Btu/lb

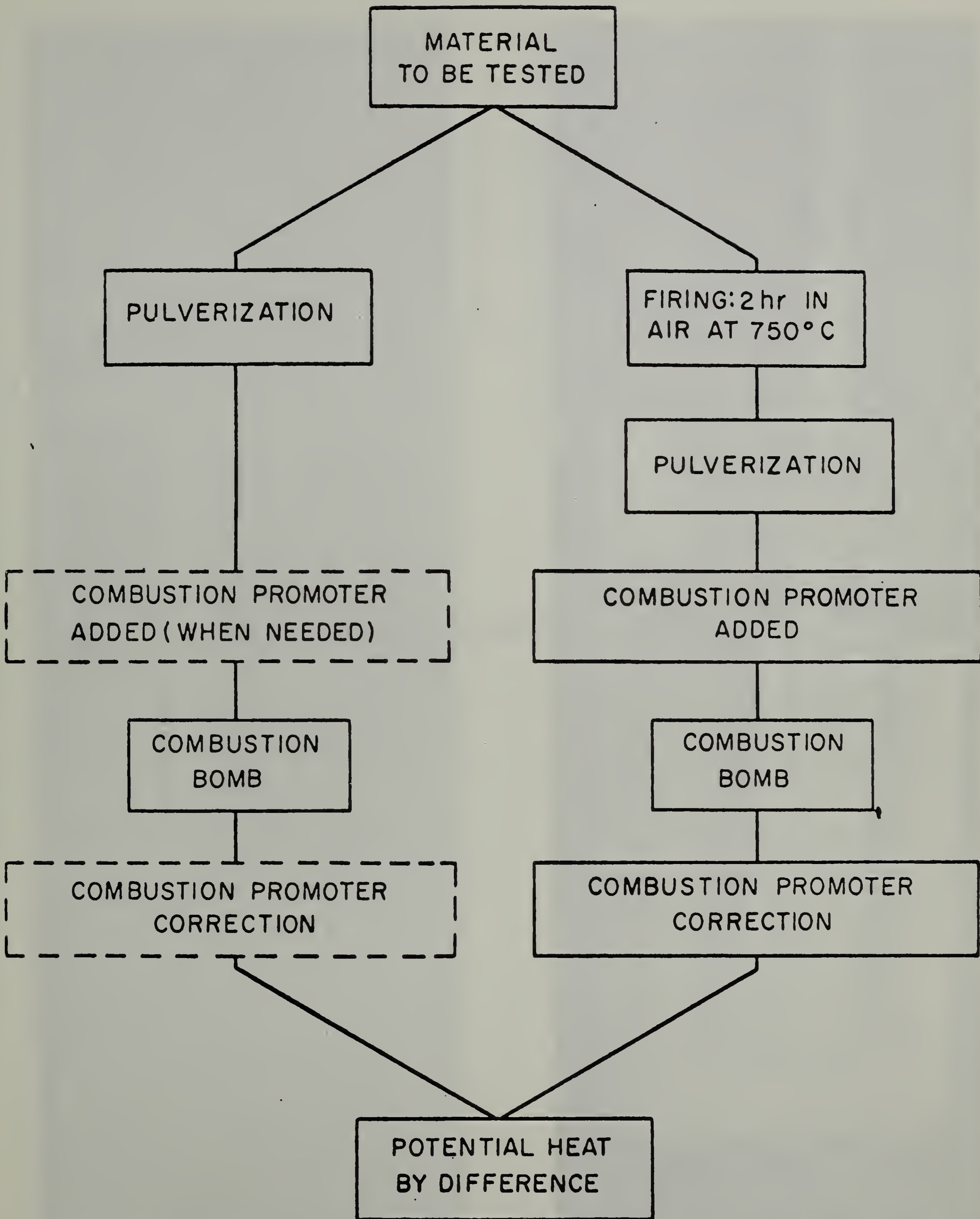

Fig. A1 Schematic Diagram of Test Procedure for
Potential Heat Measurements

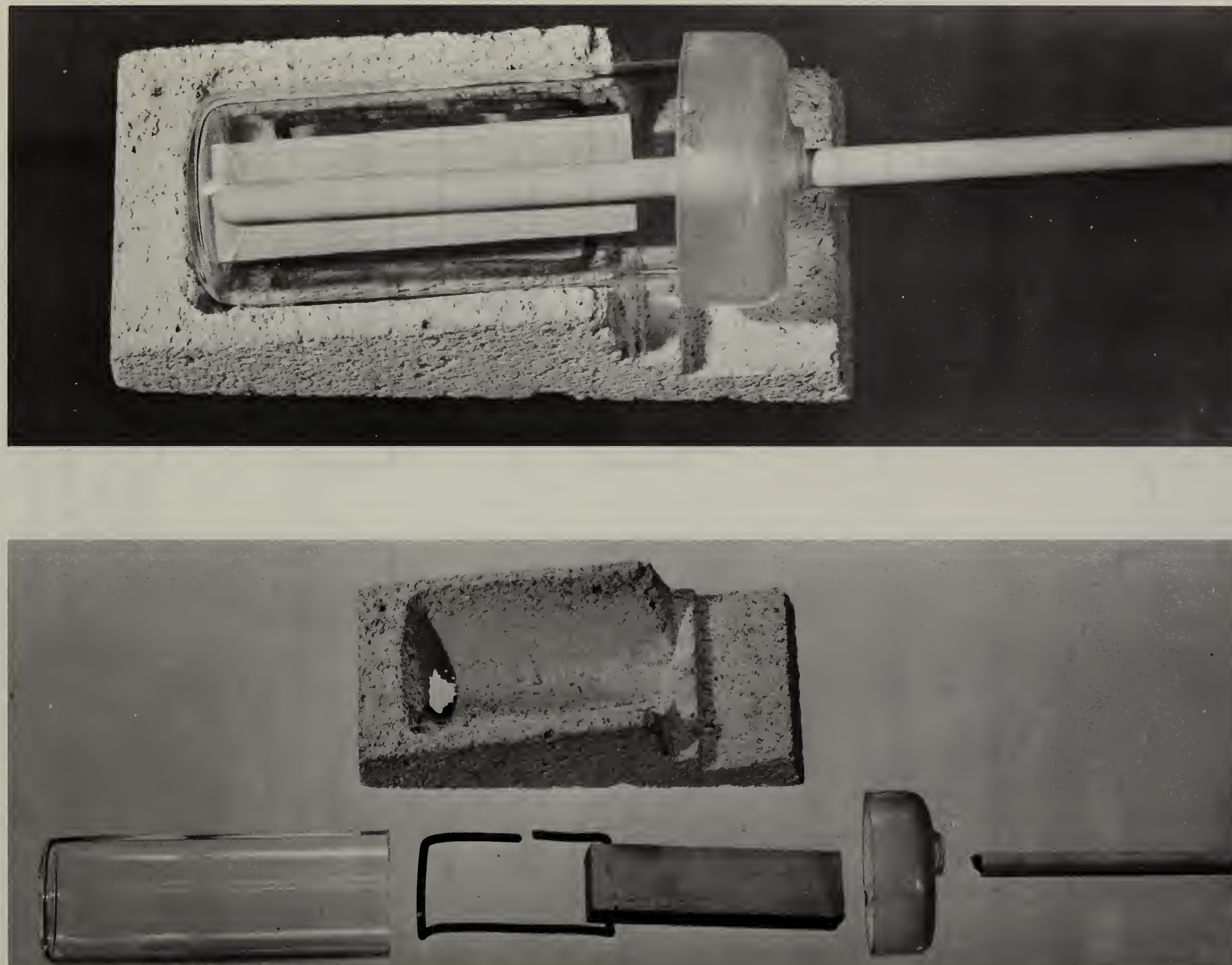

Fig. A2 Equipment for Performing Muffle Furnace Test
Top: Assembly Bottom: Component Parts

TABLE 1. TEST PLAN FOR SECOND PHASE

	MATERIAL				
	A	B	C	D	E
50% Combustion Promoter	x x	x x	x x	x x	x x
75% Combustion Promoter	x			x	

LIST OF MATERIALS

Designation	Material	Nominal Thickness in.	Measured Density lb per cu ft
A	Asbestos-cement board	1/4	121
B	Mineralized wood	7/8	24
C	Gypsum board	3/8	49
D	Mineral acoustic tile	3/4	24
E	Protected steel	1/16	281

Table 2. SUMMARY VALUES OF HEAT OF COMBUSTION AND POTENTIAL HEAT (Btu/lb)
50 % Promoter
Potential Heat = Direct Heat of Combustion - Residue Heat of Comb.X % Residue

Laboratory	Test	A Asbestos-Cement Board			B Mineralized Wood			C Gypsum Board			D Mineral Acoustic Tile			E Protected Steel		
		Direct	Residue	Pot. Heat	Direct	Residue	Pot. Heat	Direct	Residue	Pot. Heat	Direct	Residue	Pot. Heat	Direct	Residue	Pot. Heat
P	1	-207	85	-271	2364	-381	2527	-258	-246	-88	937	-264	1165	2738	2124	826
	2	-248	69	-300	1861	-299	1989	-194	-107	-120	978	-315	1250	2964	2358	859
	Avg	-228	77	-286	2112	-340	2258	-226	-176	-104	958	-290	1208	2851	2241	842
Q	1	-47	-141	63	2170	25	2158	245	-722	766	577	-279	819	3178	1812	1515
	2	-80	-173	55	2964	33	2949	146	-673	632	936	-330	1222	2859	2049	978
	Avg	-64	-157	59	2567	29	2554	196	-698	699	756	-304	1020	3018	1930	1246
R	1	32	-288	258	1899	-104	1947	-400	-733	137	965	-58	1015	2923	1651	1448
	2	-68	-207	94	2194	50	2171	-461	-839	152	1005	-4	1007	2664	1789	1074
	Avg	-18	-248	176	2046	-27	2059	-430	-786	144	985	-31	1011	2794	1720	1261
S	1	-50	51	-89	2434	142	2369	250	-627	711	966	178	814	3521	2600	1194
	2	-185	-64	-134	2355	105	2307	197	-686	699	982	90	905	3644	2524	1382
	Avg	-118	-6	-112	2394	124	2338	224	-656	705	974	134	860	3582	2562	1288
T	1	-147	24	-166	1962	-132	2024	-177	-687	325	977	8	970	3124	2345	1024
	2	-190	20	-206	2071	-183	2157	-166	-697	344	962	54	916	3194	2399	1043
	Avg	-168	22	-186	2016	-158	2090	-172	-692	334	970	31	943	3159	2372	1034
U	1	265	290	45	1237	169	1152	486	-1114	1299	653	95	570	3551	1525	2185
	2	218	808	-412	2592	-524	2828	40	630	-422	833	-38	866	3618	2092	1737
	Avg	242	549	-184	1914	-178	1990	263	-242	438	743	28	718	3584	1808	1961
V	1	-148	-133	-44	2491	82	2453	232	-684	729	1093	133	979	3479	2410	1308
	2	-320	50	-359	2351	94	2308	200	-653	677	1078	104	989	3452	2282	1398
	Avg	-234	-42	-202	2421	88	2380	216	-668	703	1086	118	984	3466	2346	1353
W	1	-135	-97	-60	2443	-135	2505	226	-631	688	1185	-64	1163	3544	2492	1309
	2	-140	-171	-6	2399	-124	2457	252	-643	723	1104	115	1082	3486	2433	1303
	Avg	-138	-134	-33	2421	-130	2481	239	-637	706	1144	26	1122	3515	2462	1306
X	1	-254	231	-434	2027	309	1883	174	-606	616	1013	145	891	3269	2456	1073
	2	-208	-84	-143	2162	61	2133	-123	-673	367	977	115	879	2745	2501	508
	Avg	-231	74	-288	2094	185	2008	26	-640	492	995	128	885	3007	2478	790
Y	1	-38	-102	43	495	-6	498	374	-337	620	923	206	746	3246	1928	1520
	2	-42	-144	72	542	23	532	458	-256	637	950	370	631	3987	2345	1878
	Avg	-40	-123	58	518	8	515	416	-296	628	936	288	688	3616	2136	1699
Z	1	190	270	-20	2375	145	2307	235	-250	418	1185	170	1039	3535	2228	1543
	2	155	255	-43	2295	165	2218	235	-190	374	1150	205	974	3420	2046	1596
	Avg	172	262	-32	2335	155	2262	235	-220	396	1168	188	1006	3478	2137	1570
Median		-118	-6	-112	2112	8	2258	216	-640	492	974	31	984	3466	2241	1288

TABLE 3. SUMMARY OF VALUES OF PERCENT RESIDUE

Material	LABORATORY											Average	Average (Phase 1)
	P	Q	R	S	T	U	V	W	X	Y	Z		
A	75.4	77.8	78.4	78.2	77.8	76.8	77.8	78.2	77.5	79.6	77.7	77.7	78.4
B	42.7	46.7	46.2	45.8	46.9	44.8	46.4	46.4	46.6	46.0	46.6	45.9	45.8
C	69.0	72.2	73.2	73.4	73.2	73.2	72.9	73.2	72.9	73.0	73.0	72.7	72.9
D	86.5	86.7	86.2	85.6	85.6	87.2	85.8	85.7	85.6	86.2	86.0	86.1	85.6
E	89.7	91.8	89.1	89.6	89.6	89.8	90.0	89.7	89.4	89.8	89.3	89.8	89.5

TABLE 4. AVERAGE VALUES OF SELECTED TEST PARAMETERS

	LABORATORY										
	P	Q	R	S	T	U	V	W	X	Y	Z
Temp. Readout, ^{o} F, assumed	.01	.005	.005	.005	.005	.04	.005	.005	.005	.01	.005
Fuse Wire Correction BTU/lb*	28	18	29	24	22	15	22	22	24	24	--
Acid Titration Correction, incl. sulphur, except Mat'l "C" BTU/lb*	16	11	30	11	7	1	9	10	8	7	17
Acid Titration Correction, incl. sulphur, Material "C" BTU/lb*	187	75	133	70	43	9	54	58	34	39	39
"Water Equivalent" of Calorimeter, $\frac{Btu}{lb\ F}$* Metal	2423		2432	2445	2419	2642	2444	2416	2454	2417	
Silica	2423	2447	2428	2461	2425		2444		2457		2508

* Values refer to a 1 gram sample. The common arrangement employed with calorimeters permits calculation of the heat of combustion in Btu per lb using samples weighed in grams. For calorimeters calibrated in calories, the values have been converted into Btu units. 1 British thermal unit (Btu) = 1055 joules (J).

TABLE 5 MEDIAN AND DEVIATIONS FROM MEDIAN

(Btu/lb)

Material	Median	LABORATORY											Median 10 Labs*
		P	Q	R	S	T	U	V	W	X	Y	Z	
						DIRECT							
A	-118	-110	54	100	0	-50	360	-116	-20	-113	78	290	-128
B	2112	0	455	-66	282	-96	-198	309	309	-18	-1594	223	2224
C	216	-442	-20	-646	8	-388	47	0	23	-190	200	19	206
D	974	-16	-218	10	0	-4	-231	112	170	21	-38	194	979
E	3466	-615	-448	-672	116	-307	118	0	49	-459	150	12	3312
						RESIDUE							
A	-6	83	-151	-242	0	28	555	-36	-128	80	-117	268	-24
B	8	-348	21	-35	116	-166	-186	80	-138	177	0	147	18
C	-640	464	-58	-146	-16	-52	398	-28	3	0	344	420	-648
D	31	-321	-335	-62	103	0	-3	87	-5	97	257	157	74
E	2241	0	-311	-521	321	131	-433	105	221	237	-105	-104	2294
						POTENTIAL HEAT							
A	-112	-174	171	288	0	-74	-72	-90	79	-176	170	80	-72
B	2258	0	296	-199	80	-168	-268	122	223	-250	-1743	4	2260
C	492	-596	207	-348	213	-158	-54	211	214	0	136	-96	560
D	984	224	36	27	-124	-41	-266	0	138	-99	-296	22	995
E	1288	-446	-42	-27	0	-254	673	65	18	-498	411	282	1274

*Lab U omitted

TABLE 6. COMPARISON BY MATERIAL RANKINGS

Potential Heat Ranking Order (Lowest = 1) (Highest = 5)	P	Q	R	S	T	U	V	W	X	Y	Z	Consensus	Median (Btu/lb)
1	A	A	C	A	A	A	A	A	A	A	A	A	- 112
2	C	C	A	C	C	C	C	C	C	B	C	C	492
3	E	D	D	D	D	D	D	D	E	C	D	D	984
4	D	E	E	E	E	E	E	E	D	D	E	E	1288
5	B	B	B	B	B	B	B	B	B	E	B	B	2258

TABLE 7 RANGE OF DUPLICATES

(Btu/lb)

Material	LABORATORY P	Q	R	S	T	U	V	W	X	Y	Z	Avg Range	Avg. Range 10 Labs*
						DIRECT							
A	41	33	100	135	43	47	172	5	46	4	35	60	61
B	503	794	295	79	109	1355	140	44	135	47	80	326	223
C	64	99	61	53	11	446	32	26	297	84	0	107	73
D	41	359	39	16	15	180	15	81	36	27	35	77	66
E	226	319	259	123	70	67	27	58	524	741	115	230	246
Avg. Range	175	321	151	81	50	419	77	43	208	181	53		
						RESIDUE							
A	16	32	81	115	4	518	183	74	315	42	15	127	88
B	82	8	154	37	51	693	12	11	248	29	20	122	65
C	139	49	106	59	10	1744	31	12	67	81	60	214	61
D	51	51	54	88	46	133	29	179	27	164	35	78	72
E	234	237	138	76	54	567	128	59	45	417	182	194	157
Avg. Range	104	75	107	75	33	731	77	67	140	147	62		
						POTENTIAL HEAT							
A	29	8	164	45	40	457	315	54	291	29	23	132	100
B	538	791	224	62	133	1676	145	48	250	34	89	363	231
C	32	134	15	12	19	1721	52	35	249	17	44	212	61
D	85	403	8	91	54	296	10	81	12	115	65	111	92
E	33	537	374	188	19	448	90	6	565	358	53	243	222
Avg. Range	143	375	157	80	53	920	122	45	273	111	55		

* Lab U omitted

TABLE 8 DIFFERENCE IN HEAT OF COMBUSTION

75% PROMOTER - 50% PROMOTER

(Btu/lb)

Material		Q	R	S	T	V	W	X	Y	Average Difference (75% - 50%)
					DIRECT					
A	75%	-253	-43	127	-12	-99	-140	-103	211	
	50%	-64	-18	-118	-168	-234	-138	-231	-40	
	Diff	-189	-25	245	156	135	-2	128	251	87
D	75%	404	1114	1074	917	1125	1633	1441	1210	
	50%	756	984	974	970	1086	1144	995	936	
	Diff	-352	130	100	-53	39	489	446	274	134
Avg.	Diff.	-270	52	172	52	87	244	287	262	111
					RESIDUE					
A	75%	-40	-198	148	-195	189	98	142	238	
	50%	-157	-248	-6	22	-42	-134	74	-123	
	Diff	117	50	154	-217	231	232	68	361	124
D	75%	-789	58	342	-273	167	217	222	547	
	50%	-304	-31	134	31	118	26	128	288	
	Diff	-485	89	208	-304	49	191	94	259	13
Avg.	Diff	-184	70	181	-260	140	212	81	310	69
					POTENTIAL HEAT					
A	75%	-222	112	13	140	-246	-217	-216	23	
	50%	59	176	-112	-186	-202	-33	-288	58	
	Diff	-281	-64	125	326	-44	- 184	72	-35	-10
D	75%	1288	1064	781	1155	982	1447	1251	742	
	50%	1020	1011	860	943	984	1122	885	688	
	Diff	268	53	-79	212	-2	325	366	54	150
Avg.	Diff	-6	-6	23	269	-23	254	219	10	70

TABLE 9 DIFFERENCE IN HEAT OF COMBUSTION

PHASE 1 (1966) - PHASE 2 (1967)

(Btu/lb)

Material	LABORATORY Q	R	S	T	V	X	Y	Z	Average
					DIRECT				
A	23	-72	43	301	-4	-96	14	-277	-8
B	-1280	316	-8	-647	45	1152	846	-30	49
C	-61	650	206	68	-80	-378	-240	-55	14
D	124	-84	60	-27	-218	37	6	27	-9
E	-573	286	-106	227	-198	187	-369	147	-50
Average	-353	219	39	-16	-91	180	51	-38	
					RESIDUE				
A	195	418	-146	6	-34	-244	-87	-357	-31
B	-150	131	-128	108	105	-222	129	-15	-5
C	155	217	87	-199	72	8	-285	-45	1
D	37	171	-26	124	-75	-289	-69	-8	-17
E	541	810	-231	-134	100	-583	-40	358	103
Average	156	349	-89	-19	34	-266	-70	-13	
					POTENTIAL HEAT				
A	-130	-400	156	297	25	114	82	1	18
B	-1211	255	52	-699	-2	1255	786	-21	52
C	-167	491	141	212	-132	-383	-27	-23	14
D	88	-231	82	-132	-153	284	66	35	5
E	-1023	-431	100	345	-280	712	-327	-185	-136
Average	-489	- 63	106	5	-108	396	116	-39	

DEPARTMENT OF COMMERCE ★ NATIONAL BUREAU OF STANDARDS ★